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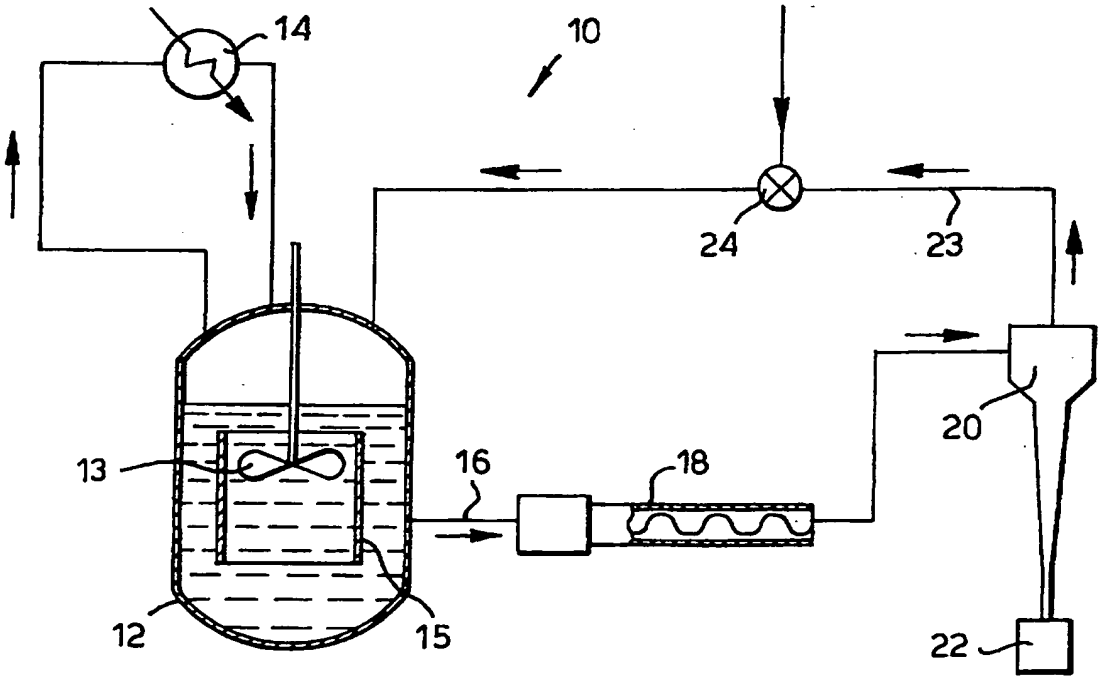
(54) Abstract Title
Reaction of bouyant solids.

(57) A chemical process plant (10) is provided for performing a chemical reaction between a first material in particulate form and a reagent in a liquid phase, the first material being less dense than the liquid and one reaction product being a solid material which is denser than the liquid. The plant (10) consists of a vessel to contain the liquid, and a recirculation duct (16, 23) communicating at each end with the vessel (12), and incorporating a pump (18) and a hydrocyclone (20). As the suspension of particles of the first material in the liquid passes through the hydrocyclone (20), the particles are subjected to shear, and the particles of the denser material are separated from the liquid and from the particles of the first material. The plant (10) is suitable for reactions involving low density metals, such as lithium, sodium or potassium, such as the reaction of lithium with butyl chloride in solution in a solvent such as hexane. The process may be batch or continuous.



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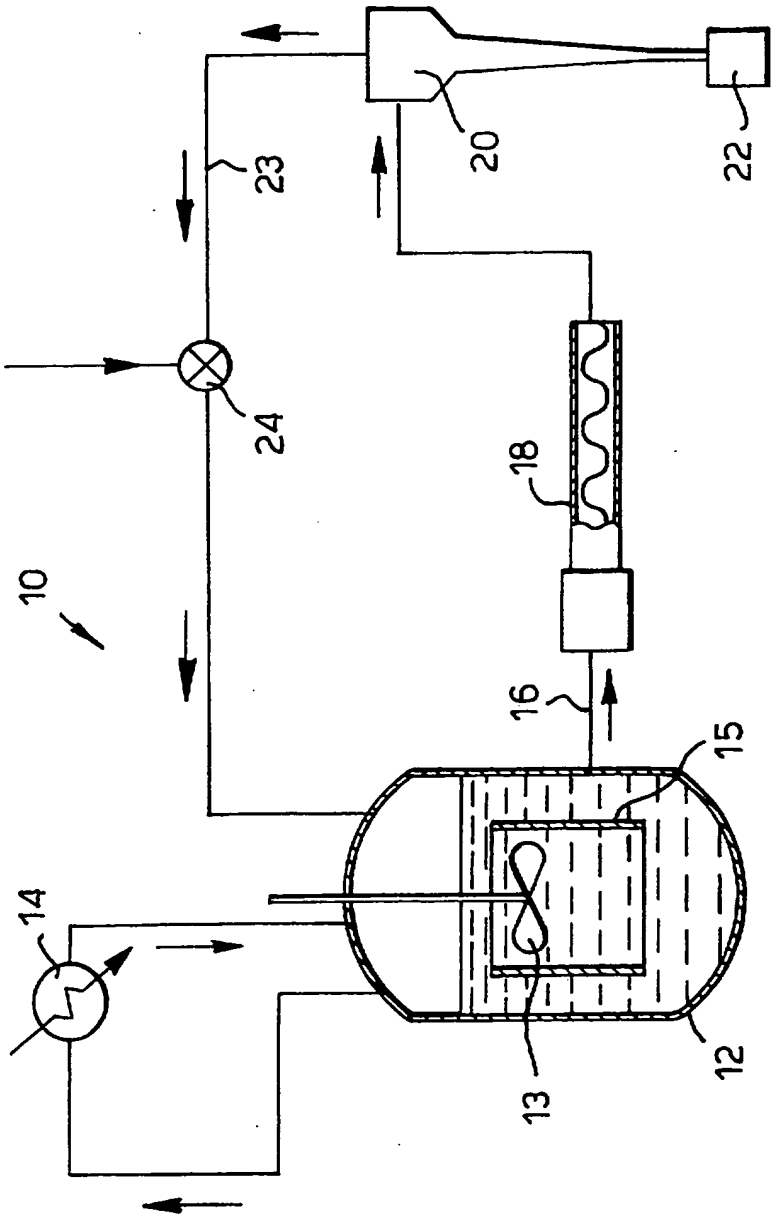
At least one drawing originally filed was informal and the print reproduced here is taken from a later filed formal copy.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Chemical Process Plant

The present invention relates to a plant for carrying out chemical processes which involve a reaction between a material in particulate form and a liquid, in which the material is less dense than the liquid, and in which a product of the reaction is a solid of higher density than the starting material.

The plant is particularly useful for reactions involving low density metals. The metals in Group I of the Periodic Table, known as the alkali metals, are highly reactive, reacting with air, and with water for example; the reactivity increases with atomic number. They are all of low density compared to most other metals, and for example lithium, sodium and potassium are all less dense than water, while rubidium has a density of only 1530 kg/m^3. The metals in Group II known as alkali earth metals have some similar properties, and in particular calcium and magnesium are both of density less than twice that of water. Chemical reactions with such metals may be performed with the metal in the form of particles in suspension in an inert liquid, the liquid acting as a solvent for a material with which the metal reacts. For example, lithium metal may be reacted with butyl chloride in solution in a solvent such as hexane. Problems can occur in such a reaction due to formation of a layer of salt on the surface of the metal.

According to the present invention there is provided a chemical process plant for performing a chemical reaction between a first material and a reagent, the reaction being carried out between the first material in particulate form and a liquid that comprises the reagent, the first material being less dense than the liquid and

one reaction product being a solid material which is denser than the liquid, the plant comprising a vessel to contain the liquid, and a recirculation duct communicating at each end with the vessel, the recirculation duct incorporating a pump and a hydrocyclone, arranged so that the liquid and the first material in suspension are circulated through the duct, so that the particles of the first material are subjected to shear within the hydrocyclone, the hydrocyclone being arranged to separate particles of the denser material from the liquid and from the particles of the first material.

The liquid may be the reagent, or the liquid may be a solution of the reagent in an inert solvent. In a preferred embodiment the recirculation duct also incorporates a mixer to introduce the reagent into the suspension of the particles of the first material in the liquid emerging from the hydrocyclone. The mixer might, for example, comprise a venturi device.

The vessel may include a stirrer, it may contain baffles or a draft tube, and may be provided with a condenser to return any liquid that evaporates back to the vessel. If the reaction is endothermic then the vessel may be provided with a source of heat, such as a steam jacket, while if the reaction is exothermic it may be desirable to provide a cooling jacket around the vessel (if the heat lost from the vessel by evaporation of the liquid is not sufficient to control the temperature).

The hydrocyclone is preferably arranged so the particles of the denser material are trapped in a container such as a grit pot.

The invention will now be further and more particularly described, by way of example only, and with reference to the accompanying drawing which shows a flow diagram for a chemical plant for producing butyl lithium.

Referring to the drawing, this shows a chemical plant 10 for performing a reaction between lithium metal and butyl chloride, so as to form butyl lithium. The plant 10 comprises a reaction vessel 12 including a stirrer 13 and provided with a condenser 14. The stirrer 13 is within an open-ended draft tube 15. A recirculation duct 16 communicates with the side of the vessel 12 and leads via a mono pump 18 to a tangential inlet of a hydrocyclone 20. The hydrocyclone 20 has two outlets: denser materials fall downwards, while lighter materials emerge from an outlet at the top of the hydrocyclone 20. Dense materials are trapped in the hydrocyclone 20 and fall into a grit pot 22, while the lighter materials flow via a duct 23 and an in-line mixer 24 such as a venturi, back to the upper part of the vessel 12. The grit pot 22 is a closed vessel connected to the lower outlet from the hydrocyclone 20, through which denser material emerges.

In use of the plant 10, the vessel 12 is initially filled with hexane liquid and lithium metal in the form of granules, pieces of wire, or pellets, and the contents of the vessel 12 are vigorously stirred by the stirrer 13 so that the particles of lithium metal, although less dense than the liquid, are distributed throughout the vessel 12. The liquid and particles of lithium are recirculated by the pump 18 through the hydrocyclone 20 and back to the vessel 12, and butyl chloride is fed into the hexane by the mixer 24. The butyl chloride reacts exothermically with the lithium metal forming the desired product (butyl lithium) but also forming lithium

chloride. The temperature of the vessel 12 may be controlled by controlling the rate at which butyl chloride is supplied. The temperature of the hexane may rise to its boiling point of 69°C, but the hexane which evaporates is returned to the vessel 12 by the condenser 14. As the particles of lithium are recirculated through the hydrocyclone 20 they are subjected to considerable shear, which ensures that lithium chloride is removed from their surfaces; and the particles of lithium chloride are trapped in the grit pot 22. It will be noted that the density of lithium metal is only 534 kg/m^3 whereas that of lithium chloride is 2070 kg/m^3, and that of hexane is 660 kg/m^3. At intervals the grit pot 22 must be emptied.

The manner of operation may be a batch process to process a batch of butyl chloride, starting with a stoichiometric excess of lithium metal. Once the batch of butyl chloride has all been added and has reacted, the liquid would be emptied from the vessel 12 and stored for use, leaving the excess lithium for the next batch. It will be appreciated that the plant 10 could be modified so that the process could be performed in a substantially continuous fashion, by continuously tapping off some of the solution of butyl lithium in hexane from the vessel 12, and continuously introducing lithium in particulate form and hexane into the vessel 12, so as to maintain a substantially constant liquid level in the vessel 12. In this case, butyl chloride would be supplied continuously to the mixer 24.

It will be appreciated that a chemical reaction plant may differ from that described above while remaining within the scope of the present invention, and that some details will depend upon the chemical reagents

involved. For example in the above reaction an alternative solvent may be used, such as cyclohexane whose density is 779 kg/m^3 and which boils at 81°C. The plant may differ, for example in having the recirculation duct 16 communicate with the vessel 12 at a different position, for example at the base of the vessel 12, or nearer the top of the vessel but just below the liquid surface. The mono pump 18 might be replaced by a different type of pump; and the venturi mixer might be replaced, for example by a fluidic vortex mixer.

Claims

1. A chemical process plant for performing a chemical reaction between a first material and a reagent, the reaction being carried out between the first material in particulate form and a liquid that comprises the reagent, the first material being less dense than the liquid and one reaction product being a solid material which is denser than the liquid, the plant comprising a vessel to contain the liquid, and a recirculation duct communicating at each end with the vessel, the recirculation duct incorporating a pump and a hydrocyclone, arranged so that the liquid and the first material in suspension are circulated through the duct, so that the particles of the first material are subjected to shear within the hydrocyclone, the hydrocyclone being arranged to separate particles of the denser material from the liquid and from the particles of the first material.

2. A process plant as claimed in claim 1 in which the recirculation duct also incorporates a mixer to introduce the reagent into the suspension of the particles of the first material in the liquid emerging from the hydrocyclone.

3. A process plant as claimed in claim 2 in which the mixer is a venturi device.

4. A process plant as claimed in any one of the preceding claims wherein the vessel includes a stirrer.

5. A process plant as claimed in any one of the preceding claims also including a condenser to return any liquid that evaporates back to the vessel.

6. A process plant as claimed in any one of the preceding claims in which the hydrocyclone is arranged to trap the particles of the denser material in a closed container.

7. A chemical process plant for performing a chemical reaction between a first material and a reagent, the reaction being carried out between the first material in particulate form and a liquid that comprises the reagent, the first material being less dense than the liquid and one reaction product being a solid material which is denser than the liquid, the plant being substantially as hereinbefore described with reference to, and as shown in, the accompanying drawing.

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Claims searched:	1 to 7	**Date of search:**	6 July 2000

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK Cl (Ed.R): B1F (F4G)

Int Cl (Ed.7): B01J 8/00, 19/18

Other: Online: EPODOC, PAJ, WPI.

Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
X	GB 1488441 (BASF AG) See whole document.	1, 4 & 6.

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.